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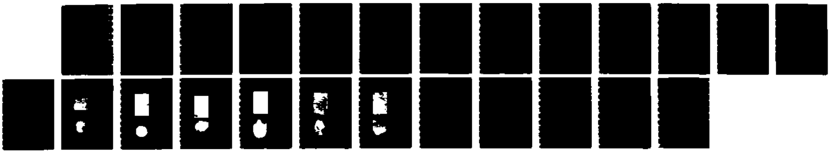

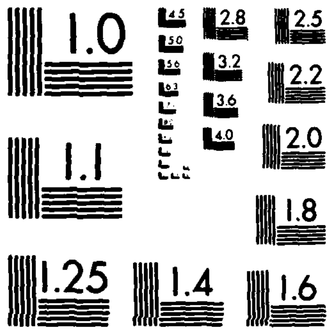
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Fiber-Matrix Interactions in Carbon-Fiber/Cement Matrix Composites

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25 July 1986

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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An understanding of the nature of the interface between fiber and composite matrix is essential for development of ceramic-matrix and metal-matrix composites. It has been shown that the use of organometallic-based coatings on carbon fibers aids processing and improves properties in metal-matrix composites. The present report deals with the use of similarly coated carbon fibers in various cement matrices. It describes the method of applying the coatings and the differences in the fiber-matrix interfacial region due to the use of different coatings. The tensile properties are

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20. ABSTRACT (Continued)

discussed and related to the differences seen at the interface as well as to differences in processability.

PREFACE

The authors would like to thank Dr. H. Katzman for his helpful technical discussions.

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FIGURES

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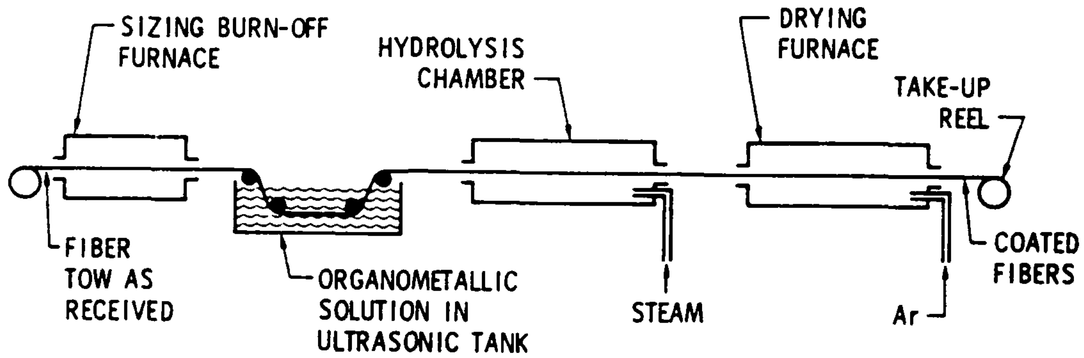
I. INTRODUCTION

There is a large and growing activity in the development of advanced fiber-reinforced ceramics and glasses for use in a variety of structural applications. The properties of these composites are influenced by the interactions between the filament and matrix materials. A strong fiber-matrix bond can result in a strong but still brittle composite, while a weak bond can result in a material with poor properties. To optimize the properties and to be able to make design trade-offs (e.g. strength vs. toughness), a more complete understanding of the interactions between the matrix and fibers is required. This report discusses the first results of our investigation into fiber-matrix interactions in a ceramic matrix system.

The purpose of this investigation is to examine the fiber-matrix interactions in a carbon-fiber/cement matrix system where an oxide coating of SiO_2, Al_2O_3, Al_2O_3-SiO_2 (3:2), or ZrO_2 has been deliberately introduced at the interface. The carbon-fiber/cement matrix system was chosen for several reasons, the most important being its potential for development of a material having a zero or near-zero coefficient of thermal expansion. This is made possible by the combination of the negative axial thermal expansion of the carbon fiber and the low modulus of the cement. Also, this system requires no elevated-temperature processing, since the cements chosen cure at room temperature. Finally, the cements can be readily modified by the addition of organometallics and/or particulates. The coatings were chosen to provide differing degrees of chemical compatibility with the matrix cements.

There are a variety of techniques available for coating carbon fibers, including chemical or physical vapor deposition, but one technique that has shown to be effective in metal-matrix composites involves coating the fiber with an oxide material 1000 to 2000 Å thick[1] by using organometallic precursors. The coating adheres to the fiber and provides a surface that the molten metal will wet during further processing. The exact coating used depends on the matrix metal desired. The organometallic is applied in an ultrasonic bath and hydrolyzed to form the oxide. It is then heat treated to drive off the organic components and consolidate the oxide. A schematic of the coating process is shown in Fig. 1. Coatings of carbides or nitrides can also be deposited by this technique.



$$Si(OC_2H_5)_4 + 2H_2O \rightarrow SiO_2 + 4C_2H_5OH\uparrow$$

$$Si(OC_2H_5)_4 \xrightarrow{\Delta} SiO_2 + 2C_2H_5OH\uparrow + 2C_2H_4\uparrow$$

Fig. 1. Schematic of Fiber Coating Process and Typical Reaction for SiO_2 Coating

II. EXPERIMENTAL PROCEDURE

Several 15-m lengths of carbon fiber tows[a] were double coated with SiO_2, Al_2O_3, SiO_2-Al_2O_3, and ZrO_2. The organometallic precursors were tetraethylorthosilicate (TEOS), aluminum isopropoxide, and zirconium n-propoxide. The precursors were applied to the tows in an ultrasonic bath, hydrolyzed in a steam bath, and heat treated to 575°C under argon. Lengths of coated and uncoated tows were cut into ~15-cm segments and infiltrated with an epoxy resin or one of two porcelain cements[b] in an ultrasonic bath. The major constituent of the cements was either zircon or silica. All of the matrices were cured at room temperature.

The tensile strengths of the various infiltrated tows were measured by attaching metal tabs to the ends of the tows, mounting the tabs in a tensile tester, and pulling the tows at a crosshead speed of 0.0042 cm/s until complete failure was obtained. The fracture surfaces were examined in a scanning electron microscope (SEM) to determine the failure modes and origins.

---

[a]T300-1K, Union Carbide Corp.
[b]Sauereisen 29 and 31, Sauereisen Cement Co.

III. RESULTS AND DISCUSSION

Fracture strengths were calculated from the breaking loads and areas were measured at the fracture origin by the SEM. The results are given in Table 1. It is evident from the data for the resin matrix materials that there has been no degradation of fiber properties as a result of the application of the coatings.

Figures 2 through 7 show the tows infiltrated with the zircon base cement for the unsized, sized, SiO_2-, Al_2O_3-, SiO_2-Al_2O_3, and ZrO_2-coated materials, respectively. From the chemical compatibility viewpoint, it would be expected that the SiO_2- and ZrO_2-coated tows would show the greatest degree of bonding with cement, while the remainder would be less well bonded. Examination of the figures leads to several observations. The first is that the fiber with no sizing or coating (Fig. 2) has very poor infiltration of the cement into the tow compared to the remaining materials. Second, the distribution of the fibers in the cement as well as the overall diameter of the infiltrated tow vary considerably with the applied coating. The best distribution and smallest diameter occur in the sized (Fig. 2), SiO_2 (Fig. 4), and ZrO_2 (Fig. 7) materials. The Al_2O_3 (Fig. 5) and Al_2O_3-SiO_2 (Fig. 6) materials showed clumping of the fibers and large diameters. A unique observation about the Al_2O_3-coated tows can be seen in the high-magnification micrograph of Fig. 5. Although each fiber is surrounded by cement, there appears to be no contact of the fiber and the matrix, i.e., there is no apparent fiber-matrix bonding. Such a condition would lead to fracture and instantaneous release of the individual fibers during tensile testing, resulting in very poor strengths. Such a result is seen in Table 1, where the strength of the Al_2O_3-coated material is the lowest of the zircon cement materials.

Table 2 compares the measured tensile strengths of the T300/zircon cement tows with the values expected from the rule-of-mixtures. The volume fraction of fibers was measured from the micrographs, and strains were estimated from the load-vs.-time chart acquired during the tensile tests. For all of the materials except the Al_2O_3-coated tows, the rule-of-mixtures appears to provide a reasonable prediction of the tensile strength. The rule-of-mixtures calculations assume complete bonding of the fiber and the matrix.

Table 1. Tensile Strength (MPa) of Various Composite Tows

Fiber	Matrix		
	Resin	Zircon	Porcelain
Unsized	---	172±46	---
Sized	3130±205	232±83	340±118
SiO_2-Coated	2680±532	111±28	416±136
Al_2O_3-Coated	3060±375	16±11	124±56
SiO_2-Al_2O_3-Coated	3090±193	45±32	218±111
ZrO_2-Coated	2780±435	87±50	110±52

Note: Unreinforced cement is ~2.8 MPa.

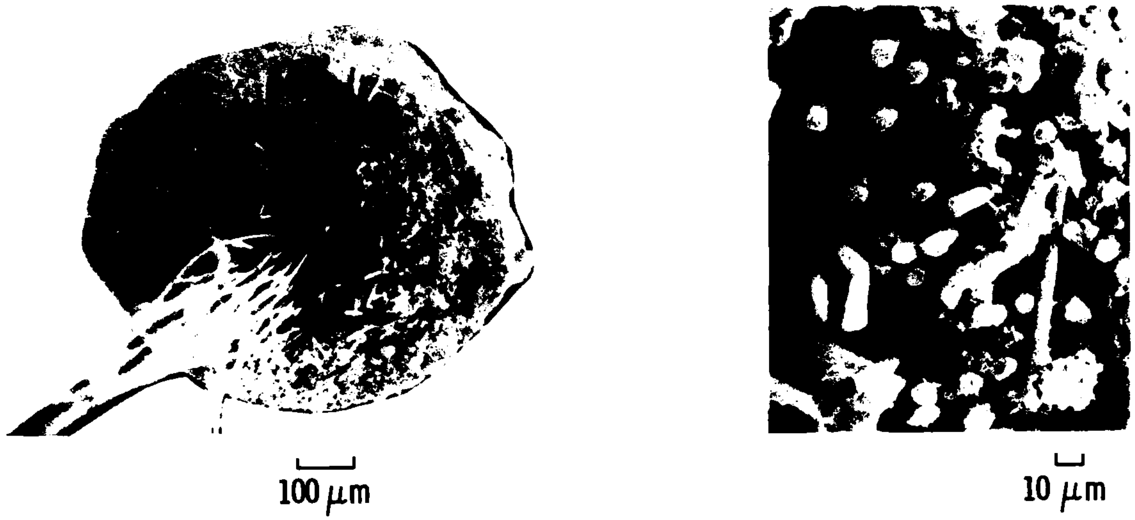


Fig. 2. Carbon-Fiber-Reinforced Cement Composite with Uncoated Fibers

Fig. 3. Carton-Fiber-Reinforced Cement Composite with Sizing Only

Fig. 4. Carbon-Fiber-Reinforced Cement Composite with SiO_2 Coating

100 μm 10 μm

Fig. 5. Carbon-Fiber-Reinforced Cement Composite with Al_2O_3 Coating

Fig. 6. Carbon-Fiber-Reinforced Cement Composite with Al_2O_3-SiO_2 Coating

Fig. 7. Carbon-Fiber-Reinforced Cement Composite with ZrO_2 Coating

Table 2. Tensile Strength of Infiltrated Tows vs. Rule-of-Mixtures Calculations

Fiber	V_F	$\epsilon_{matrix\ break}$	σ_{ROM}(MPa)	σ_{actual}(MPa)
Unsized	0.13±0.02	0.0077±0.0016	232±49	171±46
Sized	0.11±0.03	0.0093±0.0011	241±78	231±83
SiO_2-Coated	0.07±0.02	0.0063±0.0013	114±41	111±26
Al_2O_3-Coated	0.04±0.03	0.0072±00.24	75.8±59.8	16.7±11.2
SiO_2-Al_2O_3-Coated	0.04±0.02	0.0041±0.0010	42.5±25.1	45.2±32.5
ZrO_2-Coated	0.07±0.03	0.0042±0.0011	70.0±36.3	86.7±50.6

IV. CONCLUSIONS

Differences in mechanical properties in the carbon-fiber/cement matrix system which result from the presence of different coatings have been seen. The differences can be related to the ability to infiltrate the carbon tows and to the degree of bonding between the fibers and the matrix. For the zircon cement system, the rule-of-mixtures appears to provide a reasonable prediction of the tensile strengths and yields an indication of the degree of bonding enhancement due to the coatings.

REFERENCE

1. H. A. Katzman, "Carbon-Reinforced Metal-Matrix Composites," U. S. Patent #4,376,803 (15 March 1983).

LABORATORY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security projects, specializing in advanced military space systems. Providing research support, the corporation's Laboratory Operations conducts experimental and theoretical investigations that focus on the application of scientific and technical advances to such systems. Vital to the success of these investigations is the technical staff's wide-ranging expertise and its ability to stay current with new developments. This expertise is enhanced by a research program aimed at dealing with the many problems associated with rapidly evolving space systems. Contributing their capabilities to the research effort are these individual laboratories:

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Chemistry and Physics Laboratory: Atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, sensor out-of-field-of-view rejection, applied laser spectroscopy, laser chemistry, laser optoelectronics, solar cell physics, battery electrochemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, thermionic emission, photosensitive materials and detectors, atomic frequency standards, and environmental chemistry.

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